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भाग 7 फॉस्फोरस पेंटाआक्साइड का निर्धारण

(पहला पुनरीक्षण)

Indian Standard

CHEMICAL ANALYSIS OF BAUXITE

PART 7 DETERMINATION OF PHOSPHORUS PENTOXIDE

(First Revision)

ICS 71.040.40; 73.060.40

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

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Price Group 2

Methods of Chemical Analysis of Ores, Minerals and Allied Materials for Metallurgical Industry Sectional Committee, MTD 29

FOREWORD

This Indian Standard (Part 7) (First Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Methods of Chemical Analysis of Ores, Minerals and Allied Materials for Metallurgical Industry Sectional Committee had been approved by the Metallurgical Engineering Division Council.

IS 2000 : 1962 'Methods of chemical analysis of bauxite', covered the determination of different elements in bauxite. This standard was first published in 1962. The committee decided to revise this standard into different parts covering determination of each element given in IS 2000. This part covers determination of phosphorus pentoxide. The other parts are:

Part 1 Determination of loss on ignition

Part 2 Determination of silica

Part 3 Determination of alumina

Part 4 Determination of ferric oxide

Part 5 Determination of titania

Part 6 Determination of vanadium

Part 8 Determination of manganese

Part 9 Determination of magnesium and calcium

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

CHEMICAL ANALYSIS OF BAUXITE

PART 7 DETERMINATION OF PHOSPHORUS PENTOXIDE

(First Revision)

1 SCOPE

This standard (Part 7) covers determination of phosphorus pentoxide in the form of phosphomolybdic complex reduced in a constant normality sulphuric acid medium after dehydration of silica by triacid digestion by spectrophotometric method.

2 EQUIPMENT AND MATERIAL

a) *p*H meter with glass-calomel electrodes,
b) Spectrophotometer,
c) Magnetic stirrer with regulated temperature,
d) Holder for calibrated flasks,
e) Standard laboratory glasswares,
f) Electronic balance, and
g) Water bath.

3 REAGENT

3.1 Ammonium Heptamolybdate Aqueous Solution $(NH_4)_6Mo_7O_{24} . 4H_2O$ (25 g/l)

This solution shall be prepared every week.

Weigh 12.5 g of salt using the precision balance. Dissolve it without heating in 300 ml of de-ionized H_2O. Transfer into a calibrated 500 ml flask and fill up to the top line with de-ionized water.

3.2 Citric Acid Aqueous Solution $(C_6H_8O_7)H_2O$

Weigh out 50 g of acid using the precision balance. Dissolve it in one litre of de-ionized water (50 g/l).

3.3 Ascorbic Acid Aqueous Solution, 20 g/l

Prepare this solution every day.

Weigh out 2 g.of reagent using the precision balance. Dissolve it in 50 ml of H_2O.

Transfer it into a calibrated 100 ml flask and fill up to the top line with de-ionized water.

3.4 1 N Sodium Hydroxide Solution

Transfer the contents of Merck titrisol into a standard calibrated 1 litre flask. Fill up to the top line with de-ionized water. (This solution can also be prepared by dissolving NaOH in water and standardizing the solution.)

3.5 2 N Sodium Hydroxide Solution

Proceed as for the 1 N soda preparation but transfer the contents of a Merck titrisol into a standard calibrated 500 ml flasks.

3.6 Sulphuric Acid 95-97%, rd =1.84

3.7 Sulphuric Acid Solution Approximately 10 N

Carefully add 280 ml of sulphuric acid (**3.6**) to a pyrex beaker containing approximately 500 ml of de-ionized water, cool the mixture and transfer into a calibrated 1 litre flask; rinse the beaker and add the wash waters. Fill up to the top line with de-ionized water and homogenize. Cool a second time and top up the volume with de-ionized water.

3.8 Aqueous Solution Saturated with Bromophenol Blue

3.9 Diammonium Hydrogen Phosphate $[(NH_4)_2 HPO_4]$ — necessary for the plotting of the calibrated curve.

4 CALIBRATION CURVE

The colour of the reduced phosphomolybdic complex depends on the acidity of the solution. The normality of the sulphuric medium must be known. This normality is fixed at 1.2. The acidity of the determination of the samples is also fixed at 1.2 N in sulphuric acid.

4.1 Sulphuric Acid Solution Determination Approximately 10 N (*see* 3.7)

Place three 5 ml samples of the H_2SO_4 solution in a 250 ml beaker. Add approximately 150 ml of de-ionized H_2O.

Using the *p*H meter titrate it using the 2 N sodium hydroxide solution (**3.5**) until *p*H > 7. Note the volumes of sodium hydroxide used. Let V_2 be the average volume.

Calculate the normality as per the equation:

$$C_1V_1 = C_2V_2$$

or

$$C_1 = \frac{2V_2}{5} = \text{x (in normality)}$$

$$4.9\ \text{x} = \text{A g / litre of } H_2SO_4$$

4.2 Solution for 200 mg/l of Phosphorus

Weigh out 0.852 0 g of diammonium hydrogenphosphate (3.9) using a precision balance. Dissolve in de-ionized water (without heating) and calibrate to 1 litre in a standard flask.

This solution is prepared for each verification of the calibration curve.

4.3 Solution for 20 mg/l Phosphorus

Aqueous dilution to one-tenth of the volume of solution 4.2.

4.4 Solution for 1mg/l Phosphorus

Aqueous dilution to one-twentieth of the volume of the solution (4.3). This last dilution should only be made when all the reagents have been prepared and the water bath is at the correct temperature.

4.5 Plotting of the Calibration Curve

Clean with diluted hydrochloric acid a set of seven 100-ml calibrated flasks, rinse thoroughly with de-ionized water.

4.5.1 Add respectively 0-2.5-5-10-15-20-25 ml of the standard phosphorus solution (4.4).

4.5.2 Top up with de-ionized water to 60 ml for each test (from 60 to 35-ml of water measured in the test tube).

4.5.3 Using a 25-ml precision burette, add the volume $V_{H_2SO_4}$ sulphuric acid 10 N (3.7) as per the equation:

$$V_{H_2SO_4} = \frac{5.88}{A} \times 100 \times V_{H_2SO_4} \text{ (in ml)}$$

Each solution is thus of a sulphuric acid normality rigorously stabilized, 1.2 N (that is, 5.88 g H_2SO_4/l). Cool if necessary.

4.5.4 Using a pipette, drop 10 ml of citric acid solution (3.2) into each flask and homogenize.

4.5.5 Pour into each flask 12 ml of ammonium heptamolybdate solution (3.1).

4.5.6 Pour into each flask (including zero standard solution) 2 ml of the ascorbic acid solution (3.3) and shake.

4.5.7 Fill up to top line with de-ionized water and homogenize.

4.5.8 Put the 7 open calibrated flasks into a water bath of which the liquid temperature is 100 °C. Leave for 6 min.

4.6 Take the flasks out of the water bath. Cool them to ambient temperature. Fill up again to the top line with de-ionized water, if necessary, and homogenize.

4.6.1 Wait for 10 min before using the spectrophotometer in 10 mm cells at 825 nm.

4.6.2 Plot the calibration line giving the absorbance according to the amount (in mg) of phosphorus in 100 ml. Let '*K*' be the inverse of the slope of the calibration line in mg of phosphorus by variation of absorbance.

5 DISSOLUTION OF SAMPLES

Carry out digestion using 3 acids (HNO_3 –HCl – H_2SO_4) of samples (2 g for the bauxites) until white fumes appear. Leave to cool before adding approximately 200 ml of H_2O to solubilize the salts and dehydrate the silica. Separate the silica by filtration (on a white band filter), and recuperate the filtrate in the calibrated 500 ml flasks. Wash the silica with warm water and add the wash water to the filtrate (which is not topped up to the top line). Calcine the silica at 400°C and at 1 000°C, then volatilize by adding HF. The residue is melted by $KHSO_4$ and mixed with water. This aqueous mixture is added to the preceeding filtrate and is topped up with de-ionized water to the top line of 500 ml flasks.

5.1 Determination of the Acidity of Different Digestion Solutions

a) Using a pipette, put 5 ml of digestion solutions (for 2 g of bauxite is to be made upto 500 ml).

b) Add 150 ml of de-ionized water (measured in a measuring cylinder).

c) Using a dropper, pour 3 to 4 drops of the bromophenol blue solution (3.8): yellow colour.

d) Immerse the electrodes of the *p*H meter in the solution and add the 1 N soda (3.4) until the colour of indicator changes to reddish-violet. This indicator changes in a *p*H zone between 3.0 and 4.6 so as to allow only the determination of free acidity of the solutions before the precipitation of Al_2O_3 and Fe_2O_3 begins as hydroxides.

Note the volumes V_x of NaOH, 1 N (3.4) left after each determination. The concentration in g/l of H_2SO_4 is given by:

$$C_{H_2SO_4} \times \left(\frac{1 \times V_x}{5} \times 49 \right)$$

For alternate method *see* Annex A.

6 DETERMINATION OF SAMPLES

It is necessary to carry out as many blank runs tests as developed tests. For each sample, pour into each calibrated flasks.

6.1 The liquor sample to be analysed, that is *n* ml. This sample produces a quantity of H_2SO_4 equal to :

$$\left[C_{H_2SO_4} \times \frac{n}{1\,000}\right] \text{g of } H_2SO_4$$

So an amount of sulphuric acid must be added which equals:

$$5.88 - \left(C_{H_2SO_4} \times \frac{n}{1\,000}\right) = m \text{ grams of } H_2SO_4$$

So that the medium is exactly 1.2 N

The '*m*' grams are added using the sulphuric acid solution of 10 N (**3.7**).

6.2 Calculate the volume of H_2SO_4 of 10 N (**3.7**) needed to add '*m*' grams according to:

$$\left(\frac{m}{A} \times 1\,000\right) = V_{H_2SO_4} \text{ in ml}$$

6.3 Add enough water so as to obtain a volume of 60 ml in 100-ml flask.

6.4 Add the volume $V_{H_2SO_4}$ calculated (in **6.2**) using 25 ml precision burette. Shake and leave to cool if necessary.

6.5 Add 10 ml of the citric acid solution (**3.2**) and homogenize.

6.6 Using a calibrated pipette, add 12 ml of the ammonium heptamolybdate solution (**3.1**) and shake.

6.7 Add to one of the two calibrated flasks 2 ml of the ascorbic acid reducing solution (**3.3**) (developed test) and shake.

6.8 Fill up to top line with de-ionized water and homogenize.

6.9 Place the open calibrated flasks (developed and undeveloped tests) in the water bath which is at a temperature of 100 °C. Let stand for 6 min.

6.10 Leave to cool at room temperature and if necessary fill up to the top line with de-ionized water and homogenize.

6.11 Using a spectrophotometer, measure developed tests (identical conditions as for the plotting of the calibration curve), using the corresponding blank solution as reference.

7 CALCULATIONS

Let '*D*' be the different readings (in absorbance graduations) noted for each sample. Convert the absorbance values to the corresponding amounts in µg of '*P*' using the calibration curve.

The P_2O_5 concentration in the sample is:

$$\frac{K}{10^6} \times D \times \frac{500}{P} \times \frac{100}{m} \times 2.291\,4 = \%P_2O_5$$

where

P = volume (in ml) of the analyzed aliquots

m = Sample weights (2 g for bauxites)

$$2.291\,4 = \frac{P_2O_5}{2P}$$

8 INTERFERENCE

The silica interferes with phosphorus determination as they both, with the molybdate, create either a silico-molybdic complex or a phosphomolybdic complex. These two complexes are reduced simultaneously by the ascorbic acid and each gives a blue colour: as the two wave-lengths of determination are similar ($\lambda = 800$ nm of Si and $\lambda = 825$ nm for P) the elimination of silica is essential for this determination.

The acid dissolution allows the elimination of almost all the silica in the sample, nevertheless a small fraction (< 2.0 percent) of the silica passes in to the solution which can slightly enhance the P_2O_5 results.

The addition of citric acid masks the silica which has passed into the solution upto a maximum ratio of Si = 25 in the liquor sample to be developed.

ANNEX A

(*Clause* 5.1)

DETERMINATION OF THE ACIDITY OF DIFFERENT DIGESTION SOLUTIONS (ALTERNATE METHOD)

A-1 REAGENT

2.5 g ammonium metavanadate in 250 ml (1:1) HNO_3, add a solution of 40 g ammonium molybdate in 500 ml water and dilute to 1 litre.

A-2 PROCEDURE

After dissolution of the sample (*see* **5**). Take an appropriate aliquot from another solution and add 1cc. HNO_3 (conc). Boil the solution for one minute and make just ammonical with NH_4OH after adding 2.0 g NH_4Cl and one or two drops methyl red indicator, to precipitate R_2O_3. Boil and filter, wash precipitate with hot water containing little NH_4Cl and NH_4OH. Dissolve precipitate in HNO_3 and transfer to 100-ml flask and add 10 ml 0.25 percent ammonium vanadate (meta) solution and mix thoroughly and then add 10 ml of ammonium molybdate solution (5 percent), mix well and make up the volume to 100 ml with demineralised water and allow to stay for ½ an hour and measure the yellow colour density of phosphovanadomolybdate at 460 nm against a reagent blank

$$P_2O_5 = P \times 2.2914\ \%$$

A-3 PREPARATION OF AMMONIUM META VANADATE SOLUTION 0.25 PERCENT

Dissolve 1.25 g ammonium meta vanadate in 250 ml hot water and add 10 ml HNO_3 (conc) mix well, if necessary filter it. Dilute to 500 ml with demineralised water and store in polythene bottle.

A-4 PREPARATION OF AMMONIUM MOLYBDATE 5 PERCENT

Dissolve 25 g ammonium molybdate in water and make up the volume to 500 ml with distilled water store in polythene bottle.

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